Catalyst Additives to Enhance Mercury Oxidation and Capture

Quarterly Report

Period of Performance:

March 1st, 2004 through June 30th, 2004

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July 2004

DE-FC26-04NT41900

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Abstract

Methods for carrying out the carbon experiments and carbon/calcium experiments were developed. The Catalyst Test Facility (CTF) will continue to be used to derive fundamental kinetic information about each catalyst and catalyst material investigated for mercury oxidation and capture. The CTF is described and illustrated in this quarterly report. Also, the initial proposed test matrices for carbon and for SCR catalysts are presented. Future reports will show results of these experiments and also extraction of fundamental kinetic information from the data thus obtained. The results of the carbon-type comparison tests performed this quarter indicate that the same carbon types that are reactive for combustion are most active for mercury. This is likely primarily a function of the internal surface area of the carbons. As observed in the test, activated carbon and a high-surface area UBC were much more active for mercury than carbon black and soot, which have fewer active carbon sites available. It was also found that NO and SO_3 had no short-term observable impact on mercury oxidation or capture. SO_3 did slowly deactivate carbons over an extended period of time, which may be relevant for some stationary carbon catalyst applications. Additional HCl, as expected, increased the carbons activity for mercury. These are qualitative conclusions. The volume of data collected this quarter has yet to be extensively analyzed. Following a more quantitative analysis of the data taken this quarter, a detailed quantitative explanation of the findings will be presented in the report next quarter.

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Introduction

The objective of this project proposed by Southern Research Institute (SRI) is to investigate the enhancement of elemental mercury oxidation in coal-fired flue gas through catalysis. In addition to testing various catalyst materials, different catalysis implementation processes may be investigated, including duct injection of catalyst/sorbent hybrids, coated low-pressure-drop screens, and fundamental mechanisms associated with enhanced Hg-oxidation on SCR catalysts. Data obtained in this work will be provided to Niksa Energy Associates (NES) and Reaction Engineering International (REI) to develop and improve models to predict mercury speciation in full-scale boilers, burning different coal types, ranging from sub-bituminous to high-volatile bituminous coals. Where necessary, SRI will develop semi-empirical mechanistic model(s) describing the mechanisms associated with catalysis enhanced mercury oxidation. Results from the project will contribute to a greater understanding of mercury oxidation in boilers.

Executive Summary

Appropriate methods for carrying out the carbon and carbon/calcium experiments were developed. A detailed test matrix is provided in the report for these experiments. However, the experiments changed somewhat as data were collected. All of the work this quarter was carried out in the Catalyst Test Facility.

The Catalyst Test Facility (CTF) will continue to be used to derive fundamental kinetic information about each catalyst and catalyst material investigated for mercury oxidation and capture. The Catalyst Test Facility is described and illustrated in this quarterly report. At the core of the CTF is an extensive flue-gas simulation, gas flow, and metering system. The catalyst sits on a quartz frit that traverses the diameter of a 1.5" quartz reaction chamber, through which the entire gas flow must pass. The CTF simulates clean (no particles) flue gas with all the major flue-gas species present, including CO, CO_2, H_2O, O_2, N_2, HCl, NO, SO_2, SO_3, and Hg°, in concentrations that exist in the flue gases of existing power plants, burning specific coal types.

The simulated flue gas is originated from compressed gas cylinders. The gases from the cylinders are then mixed to known concentrations by use of precision mass-flow controllers. Evaporating liquid water generates the appropriate moisture content in the gas stream, and mercury is added to the system via a mercury-soaked porous-ceramic tube within an excess-volume chamber. The simulated flue-gas stream is well mixed and preheated before entering the reaction chamber. A 3"-diameter tube furnace heats the 3-ft reaction chamber, and a 1½"-tubular reactor carries the gases through the furnace and holds the catalyst samples.

The results of the carbon-type comparison tests performed this quarter indicate that the same carbon types that are reactive for combustion are most active for mercury. This is primarily a function of the internal surface area of the carbons. As observed in the test, activated carbon and a high-surface area UBC were much more active for mercury than carbon black and soot, which have fewer active carbon sites available.

It was also found that NO and SO_3 had no short-term observable impact on mercury oxidation or capture. SO_3 did slowly deactivate carbons over an extended period of time, which may be relevant for some stationary carbon catalyst applications. Additional HCl, as expected, increased the carbons activity for mercury. These are qualitative conclusions. The volume of data collected this quarter has yet to be extensively analyzed. Following a more quantitative

analysis of the data taken this quarter, a detailed quantitative explanation of the findings will be presented in the report next quarter.

Three types of SCR catalyst will be examined in the first SCR-catalyst test matrix in future quarters, plate, honeycomb, and a hybrid of the two types of catalyst. Hitachi Zosen Corp. has provided the plate catalyst. Cormetech Inc. has provided the honeycomb type catalyst, and Haldor Topsoe Inc. will provide the hybrid catalyst, which is essentially a hybrid of the plate and honeycomb geometries.

Experimental

Figures 1 and 2 show pictures of the CTF's quartz furnace (micro-reactor), gas-conditioning bubblers for mercury speciation and stabilization prior to mercury monitoring, flue-gas continuous emission monitors (CEMs), and gas-flow control systems. Both elemental and total mercury are measured at the outlet of the CTF. The CTF simulates clean (no particles) flue gas with all the major flue-gas species present, including CO, CO_2, H_2O, O_2, N_2, HCl, NO, SO_2, SO_3, and Hg^o, in concentrations that exist in the flue gases of existing power plants, burning specific coal types. The simulated flue gas is originated from compressed gas cylinders. The gases from the cylinders are then mixed to known concentrations by use of mass flow controllers. Evaporating liquid water generates the appropriate moisture content in the gas stream, and mercury is added to the system as elemental mercury vapor carried in a clean air stream. The simulated flue-gas stream is well mixed and preheated before entering the reaction chamber. A 3"-diameter tube furnace heats the reaction chamber, and a 1½"-tubular reactor carries the gases through the furnace and holds the catalyst samples.

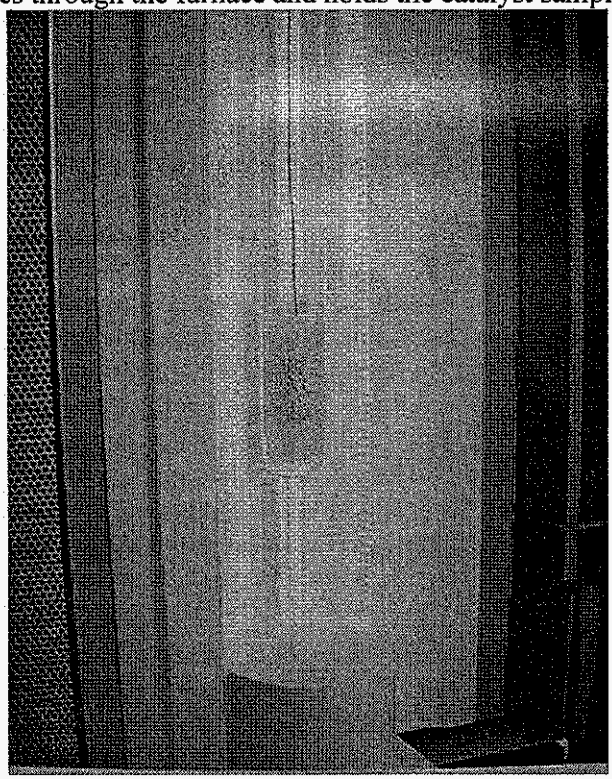

Figure 1. CTF quartz furnace with catalyst at ~1000 °C.

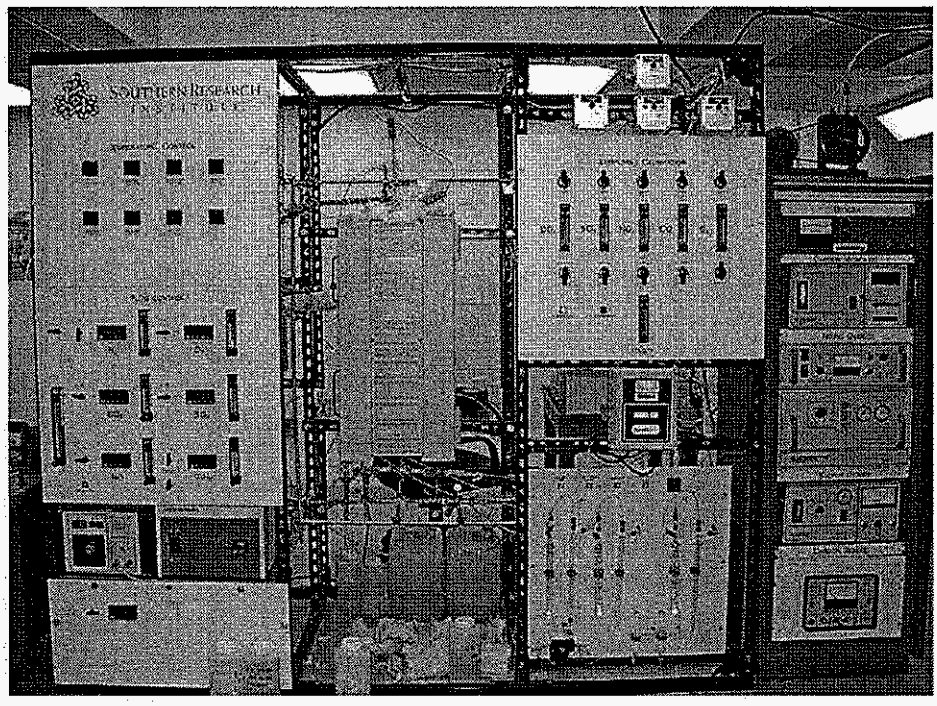

Figure 2. CTF furnace, gas-injection system, flue-gas CEMs, and Hg gas-conditioning system.

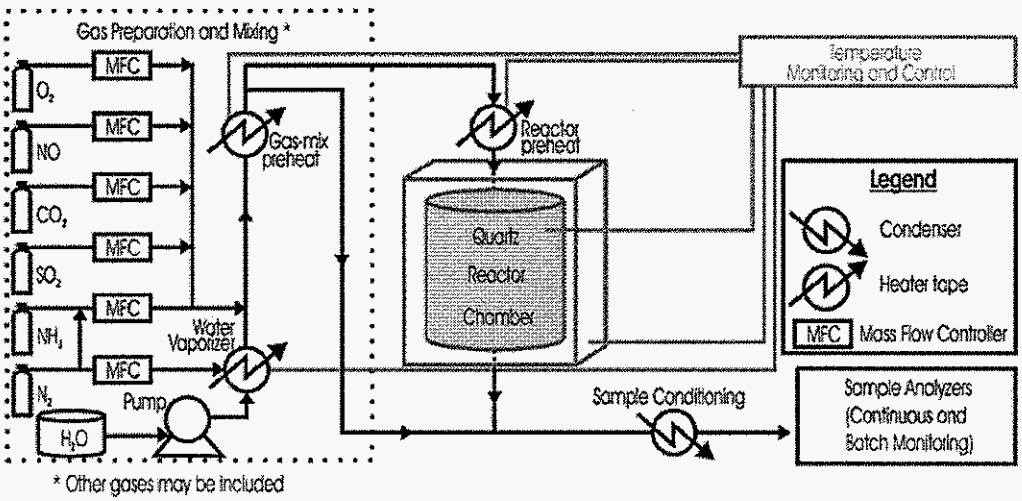

Figure 3. CTF gas flow system.

All heated sections of the micro-reactor within the CTF system are made of quartz glass to limit side reactions that might occur as a result of wall effects. A semi-continuous emission monitor (SCEM) is employed to detect the mercury levels exiting the reaction chamber. A gas-conditioning system is used to convert all Hg into the elemental form, for detection using a combined gold-trap and atomic fluorescence monitor. A Tekran Model 2573A Mercury Vapor Analyzer is used to detect the elemental mercury. Along with mercury, simultaneous

measurements of oxygen, carbon dioxide, nitrogen oxides, and sulfur dioxide are made using continuous emission monitors. Figure 3 shows a schematic of the CTF system layout.

The CTF system (see Fig. 3) was characterized for proper mercury delivery and side reactions inherent in the system. The mercury delivery system used for this project was an excess-volume chamber containing mercury within a porous ceramic tube, located at the bottom of the chamber. The amount of mercury exiting the spiking device is determined by the temperature, pressure, and flow rate of air passing through this mercury chamber. The delivery rate of mercury was confirmed by collecting the output by wet chemistry and detection by analytical testing. This was done directly from the output of the Hg-spiking device, thus eliminating any biasing from the rest of the system. Once the delivery rate was confirmed, the micro-reactor portion of the CTF system was characterized for side reactions. This was performed in two steps, first using an inert stream of nitrogen and then a simulated flue-gas stream. In each case, the source mercury stream was injected into the larger stream of gas in the micro-reactor, and recoveries were measured. During inert injection of 10 $\mu g/Nm^3$ of mercury, a recovery of 10 +/- 0.5 $\mu g/Nm^3$ of mercury was measured. Of the total mercury recovered, >95% was elemental mercury. During simulated flue-gas injection, the level of mercury recovered was also 10 +/- 0.5 $\mu g/Nm^3$. However, of the total mercury recovered, the elemental mercury fraction was only ~75%. The simulated flue-gas conditions are provided in Table-1. The total system flow rate for this gas was 5 lpm (dry) at 70 °F and 1 atm.

Table 1. Simulated flue-gas composition.

Simulated Flue Gas Concentrations	
Gas Component	*Concentration*
➢ Water Vapor	10 vol%
➢ Oxygen	6 vol % - Dry
➢ Carbon Dioxide	15 vol% - Dry
➢ Sulfur Dioxide	500 ppmv - Dry
➢ Hydrogen Chloride	2 ppmv - Dry
➢ Nitrogen Oxide	300 ppmv -Dry
➢ Mercury	10 $\mu g/m^3$ - Dry
➢ Nitrogen	Balance - Dry
Total	**100 vol% Dry**

In addition to the above characterization and validation of the CTF system, a background mercury baseline was performed for the temperature range across which the catalyst experiments were to be performed. A blank reactor (containing no catalyst) was inserted into the furnace. The flow rates and gas composition were set to those shown in Table-1. Six different temperatures were consecutively examined, specifically 260 °C, 316 °C, 371 °C, 427 °C, 482 °C, 538 °C (500 °F, 600 °F, 700 °F, 800 °F, 900 °F, and 1000 °F) (see Fig. 4). As shown in Fig. 4, the total and elemental mercury steady-state concentrations in the simulated flue gas were unaffected by temperature. An initial spike of mercury was observed each time the temperature was increased, but this transition quickly (<10 minutes) returned to a steady-state level similar to previous steady-state conditions obtained at lower temperatures.

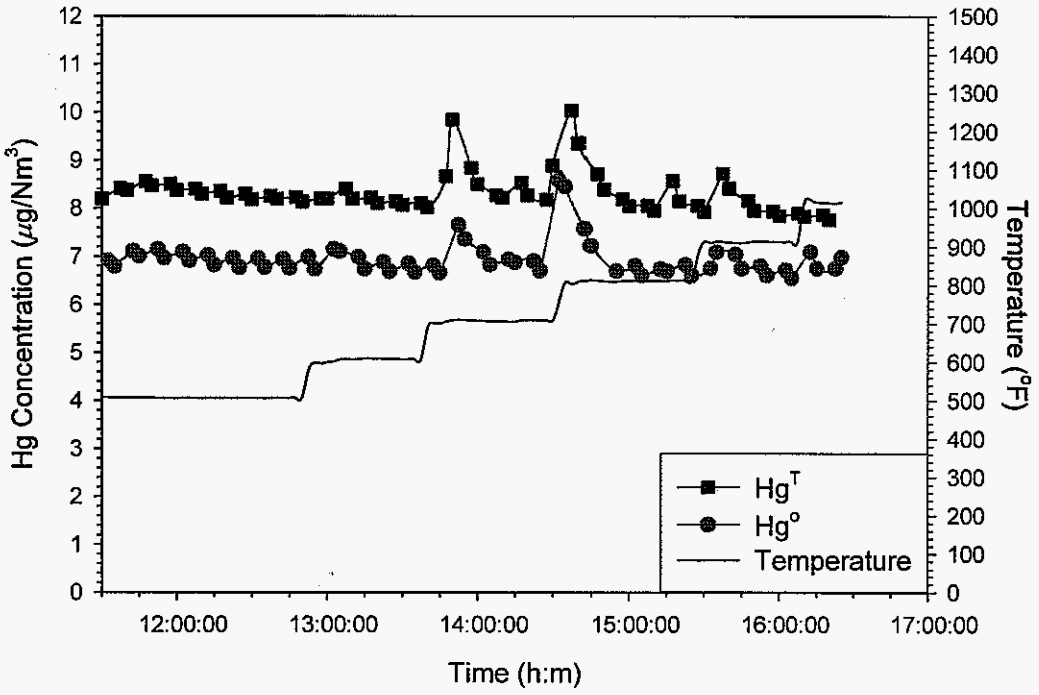

Figure 4. Background.

For additional details concerning the validation and operation of the catalyst test facility, refer to the March 04 DOE Quarterly Report for the Calcium-Based Hg-Sorbents project conducted at Southern Research Institute [1].

The flow rates, temperatures, and concentrations were continuously monitored and maintained throughout the test program. Catalyst samples were put into the quartz reactor with a quartz frit and quartz filter paper to prevent the sample from contaminating the gas-flow system. The bed depth of the catalysts was approximately one-third of an inch in the direction of gas flow. The precise bed depth was measured for each test. At the beginning of each experiment, a blank quartz reactor was inserted into the gas stream to collect baseline data. After sufficient data were collected, the blank reactor was removed and immediately replaced with the catalyst-packed reactor. The reactor was allowed to come to the initial 149 °C (300 °F) temperature, at which time the exposure experiment began. Subsequently, higher temperatures were investigated, up to 594 °C (1100 °F). At the conclusion of each experiment, the quartz filter paper and exposed sample was disposed of and the quartz reaction chamber was thoroughly cleaned and repacked with the next catalyst to be tested. These steps were precisely repeated for each catalyst.

Results and Discussion

Carbon Catalyst Tests. Table-2 contains the parameters that were adjusted during the experimental matrix presented in Table-3. Four types of carbon were examined in this first test matrix, unburned carbon (UBC), FGD activated carbon (AC), carbon black (CB), and soot. The UBC was obtained from separating the residual carbon from bituminous-coal flyash. Bituminous-coal flyash is generally higher in carbon than PRB flyash, and the UBC is primarily

contained in separate carbon particles, whereas PRB-flyash UBC is generally intimately associated with its flyash mineral matter. Coal-blending and ash-injection investigations previously conducted at Southern Research Institute (SRI) have shown that UBC in ash has a significant effect on both catalytic oxidation of mercury and catalytic capture of mercury by calcium [1-6]. In fact, it was found in the previous work that UBC enhances the capture of both elemental and oxidized mercury in the presence of calcium in PRB flyash and in calcium-based sorbents [4-6]. Hence, enhancement of Hg-oxidation and capture by different forms of carbon on several different forms of calcium were also be examined in this test matrix.

Activated carbon was used as a comparative standard of mercury removal as well as a representative fine-pore structure carbon. Carbon black and soot will provide several other different types of carbon for comparison in terms of sorptive and catalytic activity. Soot is created during conditions of poor combustion in the furnace and to some extent during firing of some retrofit low-NO_x technologies, such as low-NO_x burners. Carbon black is more graphitic in nature than activated carbon.

Soot carbon may have a different reactivity than UBC, in terms of mercury sorption and catalytic activity for enhancing mercury oxidation and capture. One of the purposes of including soot in the test matrix is to quantitatively determine how much different soot is than UBC. This information may then be used to design strategies to optimize mercury oxidation and/or removal through combustion modifications.

Table 2. Parameters examined.

Carbon Type	**Simulated Flue Gas Type (SFGT)**
1. UBC from bituminous coal ash	1. PRB coal
2. FGD Activated Carbon	2. Low-sulfur bituminous
3. Carbon Black	3. Higher-sulfur bituminous
4. Soot	4. Isolation of NO, HCl, and SO_3
Temperature (°F)	**Mineral Matter Mix (MMM)**
1. 300	1. 100% hydrated lime
2. 500	2. 80% hydrated lime
3. 700	3. 90% hydrated lime
4. 900	
5. 1100	

The initial tests performed in the matrix shown in Table 3 provided information that was used to further define tests toward the end of the test matrix. Question marks (?) were placed in Table 2 to reflect that a decision as to the specific parameter setting depended on the information learned during the performance of the initial tests in the matrix. In some cases, "2 or 3" was given to reflect a narrow constraint on the carbon type, but a decision as to whether carbon type "2" or "3" was used depended on results obtained from the first part of the test matrix.

Item "4" under simulated flue gas type (SFGT) was isolation of NO, SO_3 and HCl. This was done by establishing a SFGT of either 1, 2, or 3, followed by injection of HCl and SO_3 independently. Hence, condition "4" was not presented alone, but as #,4 (i.e., 1,4 means that SFGT-1 was established, followed by independent injection of HCl and SO_3 in separate tests).

Table 3. Benchscale experimental matrix.

Condition Number	Carbon Type	Temp.	SFGT	MMM
1	2	1	1	---
2	2	2	1	---
3	2	3	1	---
4	2	4	1	---
5	2	5	1	---
6	2	1	2	---
7	2	2	2	---
8	2	3	2	---
9	2	4	2	---
10	2	5	2	---
11	3	1	1	---
12	3	2	1	---
13	3	3	1	---
14	3	4	1	---
15	3	5	1	---
16	3	1	2	---
17	3	2	2	---
18	3	3	2	---
19	3	4	2	---
20	3	5	2	---
21	2 or 3	1	3	---
22	2 or 3	2	3	---
23	2 or 3	3	3	---
24	2 or 3	4	3	---
25	2 or 3	5	3	---
26	2	1	1, 4	---
27	2	?	1, 4	---
28	3	1	1, 4	---
29	3	?	1, 4	---
30	1	1	1	---
31	1	?	1	---
32	1	1	?	---
33	1	?	?	---
34	4	1	1	---
35	4	?	1	---
36	4	1	?	---
37	4	?	?	---
38	2 or 3	1	1	1
39	2 or 3	1	1	2
40	2 or 3	1	1	3
41	2 or 3	2	1	?
42	2 or 3	3	1	?
43	2 or 3	4	1	?
44	2 or 3	5	1	?

The CTF at Southern Research Institute (SRI) was used to conduct all benchscale experiments in this work. The quartz reactor will be filled with 3 grams of sample in a packed bed for each test. The samples will be compared on a mass basis rather than on a surface area basis because surface area is considered an inherent property and difference of each carbon type, and because practical application of these carbons will be compared on a cost/mass basis. Furthermore, the test method used to compare carbon types involved significant transport limitations. Transport limitations will also be a significant factor in practical application of these carbon catalysts in full-scale boiler systems. The packed bed and flow rates through the reactor are designed to lessen the dominance of capillary effects. In order to avoid the misleading information obtained from other benchscale apparatus, where long residence times allowed acid buildup to significantly alter sample properties, each test condition used a fresh sample. Two identical quartz reactors were used to allow a quick change between test conditions. At each test-condition change, the quartz reactor was switched out with a fresh reactor setup. The initial stable mercury measurements were used for each test condition, before significant acid gas condensation or carbon conversion had a chance to alter the catalyst properties.

A large volume of data was generated in this quarter on different carbon types. There was not sufficient time in this quarter necessary to perform the detailed and thorough analysis required for complete extraction of all the information that the data contains. Hence, a significant portion of next quarters efforts on this project will be to further analyze the data taken this quarter. Therefore, only limited results from the testing this quarter are presented in this

quarterly report. However, there were some significant observations made from the data taken this quarter that are discuss here.

The isolation experiments of HCl, SO_3, and NO indicated that NO and SO_3 have limited short-term effects on the activity of the carbons and carbon/calcium mixtures toward mercury. It was observed that after an hour of testing the carbons were somewhat deactivated by the presence of SO_3. This would be important for stationary catalysts or baghouse filters in a COHPAC or TOXICON configuration. Other than that, the presence or absence of SO3 and NO had no impact on mercury oxidation or capture by carbons or carbon/calcium mixtures. The impact of HCl was significant however, mercury oxidation and removal increasing with increasing concentration of HCl. A more quantitative analysis and presentation of these results will be provided in next quarters report on this project.

The preliminary results of mercury removal on the four carbon types investigated are presented in Figs. 5-7. Figure 5 shows a graph for activated carbon, Fig. 6 for unburned carbon (UBC), and Fig. 7 for carbon black and soot. As shown, activated carbon and the UBC tested were somewhat similar in activity for mercury. The UBC used was of a high UBC bituminous-coal fired power-plant ash. The relatively high UBC in the ash suggests that most of the carbon was of a relatively young and reactive char with a relatively high surface area. This type of UBC is expected to be much more reactive than UBC from a mature char that has been mostly burned out [7, 8]. The result of the measured internal surface area of the UBC tested is pending and will be presented in the next quarterly report.

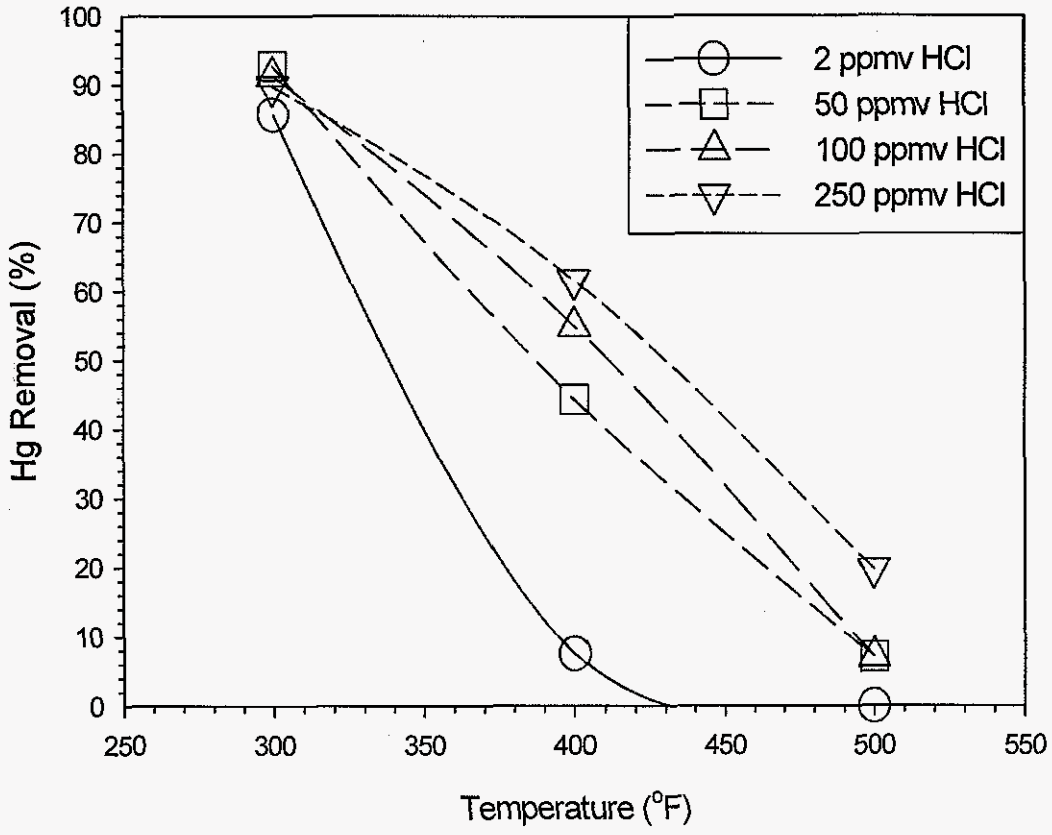

Figure 5. Activated carbon.

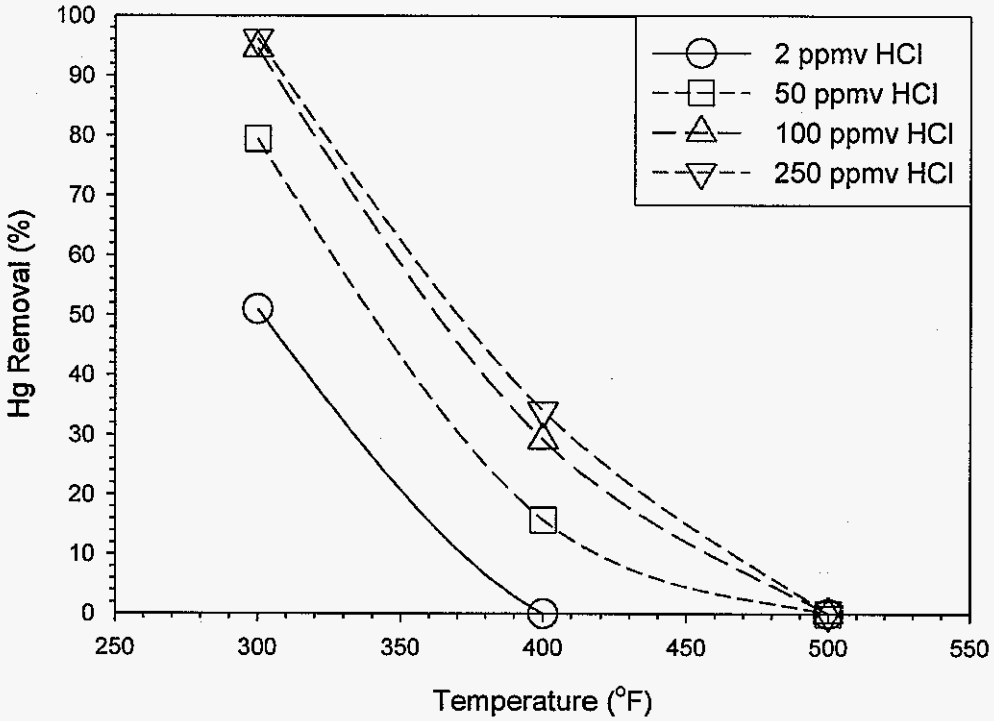

Figure 6. Unburned carbon (UBC).

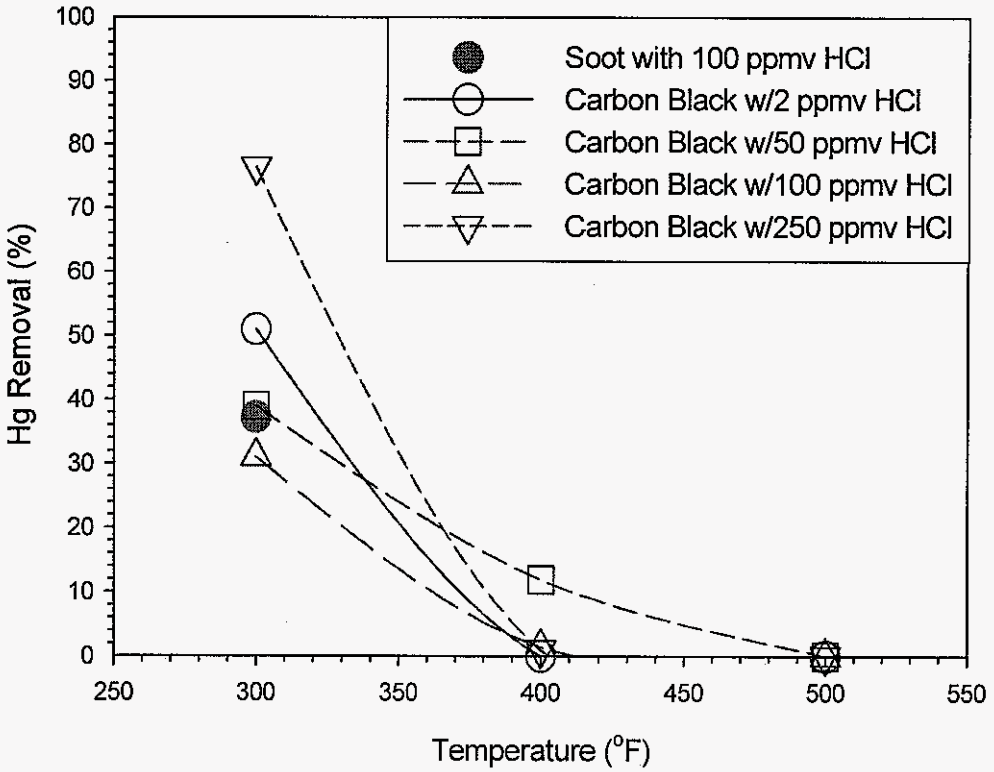

Figure 7. Carbon black (loose black) and acetylene soot.

The activity of carbon black and soot are less then that of UBC or activated carbon. Carbon black is much more graphitic, with much less surface area than UBC or activated carbon. The soot tested was made from acetylene, and hence is not the same as coal soot. However, coal soot, like acetylene soot, is less reactive with oxygen than typical high surface area chars. Although the soot produced has a lot of bulk porosity, the number of available active sites are few. This lack of soot activity with oxygen is consistent with the lack of activity with mercury. It appears that carbon types that have a high combustion rate will also have a high activity with mercury. Hence, it is not just the amount of UBC that is important, it is the type of UBC as well.

Test Matrix for SCR Catalysts. Table-4 contains the parameters to be adjusted during the experimental matrix presented in Table-5 for examination of SCR catalysts to enhance mercury oxidation. This test matrix on SCR catalysts is to be performed during future quarters.

Table 4. Parameters to be examined.

Catalyst Type	**Temperature (°F)**
1. Plate (Hitachi Zosen Corp.)	1. Low*
2. Honeycomb (Cormetech Inc.)	2. Mid*
3. Hybrid (Haldor Topsoe Inc.)	3. High*
Simulated Flue Gas Type (SFGT)	**Measurements**
1. PRB coal	1. SO_2/SO_3 Conversion
2. Low-sulfur, low-chlorine, bituminous	2. NO/NO_2 outlet concentrations
3. Higher-sulfur & chlorine bituminous	3. Mercury Removal
4. Isolation of HCl, and SO_3, and NH_3	4. Mercury Oxidation
	5. Complete gas composition, flows, etc.

*Actual temperatures to be determined with input from catalyst suppliers.

Table 5. Benchscale experimental matrix.

Condition Number	Catalyst Type	Temp.	SFGT	Meas.
1	1	1	1, 4	1-5
2	1	2	1, 4	1-5
3	1	3	1, 4	1-5
4	1	1	2, 4	1-5
5	1	2	2, 4	1-5
6	1	3	2, 4	1-5
7	2	1	1, 4	1-5
8	2	2	1, 4	1-5
9	2	3	1, 4	1-5
10	2	1	2, 4	1-5
11	2	2	2, 4	1-5
12	2	3	2, 4	1-5
13	3	1	1, 4	1-5
14	3	2	1, 4	1-5

Condition Number	Catalyst Type	Temp.	SFGT	Meas.
15	3	3	1, 4	1-5
16	3	1	2, 4	1-5
17	3	2	2, 4	1-5
18	3	3	2, 4	1-5
19	1	1	3, 4	1-5
20	1	2	3, 4	1-5
21	1	3	3, 4	1-5
22	2	1	3, 4	1-5
23	2	2	3, 4	1-5
24	2	3	3, 4	1-5
25	3	1	3, 4	1-5
26	3	2	3, 4	1-5
27	3	3	3, 4	1-5

Three types of SCR catalyst will be examined in this first test matrix, plate, honeycomb, and a hybrid of the two types of catalyst. Hitachi Zosen Corp. will provide the plate catalyst.

Cormetech Inc. will provide the honeycomb type catalyst, and Haldor Topsoe Inc. will provide the hybrid catalyst, which is essentially a hybrid of the plate and honeycomb geometries.

The initial tests performed in the test matrix shown in Table 5 will provide information used to define tests for additional test matrices. Item "4" under simulated flue gas type (SFGT) is isolation of SO_3, HCl, and NH_3. This will be done by establishing a SFGT of either 1, 2, or 3, followed by injection of HCl, SO_3, or NH_3 independently. Hence, condition "4" is not presented alone, but as #,4 (i.e., 1,4 means that SFGT-1 will be established, followed by independent injection of HCl, SO_3, and NH_3 in separate tests).

The CTF at Southern Research Institute (SRI) will be used to conduct all benchscale experiments in this work. The basis of comparison will be based on specific NO_x removals and SO_3 levels desired. Hence, the catalyst vendors have or will provide catalysts formulated for a given NO_x removal in a specific flue-gas environment. The amount of each catalyst used for these comparative tests on Hg oxidation will be determined experimentally at the beginning of the test matrix, based on NO_x removal. After the amount of each SCR catalyst has been established to obtain identical NO_x removals, then these catalyst amounts will be used in the test matrix outlined in Table 5 to examine the relative potential of each catalyst to oxidize mercury. The generation of SO_3 will also be examined.

While SCR catalysts obtain an equilibrium value with respect to NO_x reduction fairly quickly, much longer times are required to obtain equilibrium with respect to mercury oxidation. Therefore, it is anticipated that each test condition will take 24 hours to operate. The doping of SO_3 and/or HCl may also have a long equilibration time with respect to mercury oxidation, which would similarly take additional time. While 36 test conditions are listed in Table 5, each condition really represents 4 test conditions, including a baseline without catalyst, the with-catalyst baseline, and additional conditions with SO_3, HCl, and NH_3 separately injected.

The test matrix presented in Table 5 will take between 8 and 24 weeks to conduct, depending on the necessary equilibration times.

Conclusions

Four different carbon types were tested for Hg-oxidation and removal potential, activated carbon, carbon black, unburned carbon from coal ash, and acetylene soot. The results of these tests indicate that the same carbon types that are reactive for combustion are most active for mercury. This is primarily a function of the internal surface area of the carbons. As observed in the test, activated carbon and a high-surface area UBC were much more active for mercury than carbon black and soot, which have fewer active carbon sites available.

It was also found that NO and SO_3 had no short-term observable impact on mercury oxidation or capture. SO_3 did slowly deactivate carbons over an extended period of time, which may be relevant for some stationary carbon catalyst applications. Additional HCl, as expected, increased the carbons activity for mercury. These are qualitative conclusions. The volume of data collected this quarter has yet to be extensively analyzed. Following a more quantitative analysis of the data taken this quarter, a detailed quantitative explanation of the findings will be presented in the report next quarter.

A detailed test matrix for testing the three commercial SCR catalysts described in the report has been presented, and work will begin on these tests next quarter.

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